Top Loaded $TM_{01\delta}$ Mode Cylindrical Dielectric Resonator for Complex Permittivity Characterization of Liquids

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Abstract. *This paper reports on a technique of using a very high quality factor cylindrical dielectric resonator operating in $TM_{01\delta}$ mode to characterize liquids available in trace quantities. The proposed measurement technique is based on the resonant perturbation theory and the resonator is used as a sensor to determine complex permittivity of solvents at 10.5 GHz. Owing to a very high unloaded quality factor of about 5000 the proposed sensor can characterize low to medium loss materials with high accuracy. Small quantities of isopropanol and ethanol have been characterized to compute their solution ratio when mixed with different quantities of distilled water. Low cost and versatility of the proposed structure make it a prime choice for pharmaceutical industry where high standards of purity need to be ensured.*

Keywords

Material characterization, complex permittivity, cylindrical dielectric resonator, resonant perturbation techniques

1. Introduction

In recent years material characterization at microwave frequencies has gained much attention due to its rapid growing demand in different industries. Based on the dielectric properties each material has a unique signature when it is exposed to electromagnetic fields. Material properties such as permittivity, permeability and conductivity are the ones which help us in determining the behavior of materials. Among these material properties, permittivity of the material is an important attribute that gives details on the electrical nature of a material. For this purpose, several dielectric characterization techniques have been presented in literature [1], [2]. The choice of a specific technique is based on various factors such as the required degree of accuracy, nature of material, application of material, etc. Resonant perturbation techniques are considered more accurate and sensitive for calculating complex permittivity of materials [2]. Among various material categories, characterization of liquids is very important due to their vast applications in pharmaceutical and chemical industry [3]. For characterization of liquids, Substrate Integrated Waveguide (SIW) sensor based on the cavity perturbation theory has been proposed earlier [4], [5]. SIWs are planar and highly sensitive to a small change in cavity but their realization is complex. The solution proposed in [5] has addressed the complexity of creating vias by replacing them with metalized channels on the boundary walls however, these structures have low unloaded quality factor of about 58. Material characterization through microstrip resonator has been reported in [6], [7], the techniques proposed are planar and require small quantity of liquids but they are less sensitive and have low quality factor. Researchers have also proposed measurement cells based on open Whispering Gallery Mode (WGM) Dielectric Resonators (DR). This choice is due to the fact that DRs have very high quality factors due to which high accuracy of measurement can be achieved; furthermore the open structures placement of materials is easier. The open WGM resonator proposed in [8] has DR made of Material Under Test (MUT) which is excited by rectangular dielectric waveguides. This technique is only suitable for low loss materials. Another WGM resonator technique proposed in [9] characterizes the material by putting the MUT in the form of droplet on top of DR. For liquids in trace amounts and the ones with fast evaporation rate this technique is not effective. The WGM resonator proposed in [10] and [11] uses microfluidic channel to characterize the liquids. The proposed technique has high quality factor and is sensitive to small changes of MUT but it requires challenging creation of microfluidic channels. There is an increasing need of a sensor for material characterization which is easier to implement, that has high quality factor with no conductor losses and which allows easy loading of MUT.

In this paper a very sensitive top loaded $TM_{01\delta}$ mode Cylindrical Dielectric Resonator (CDR) is proposed as a sensing element which operates at a frequency of 10.5 GHz. It offers a resonant frequency shift of 6.6 MHz for a change of unity in dielectric constant of the MUT. The sensor is compact and easily realizable with a very high quality factor. The dielectric characterization of iso-

propanol and ethanol has been carried out and it is demonstrated that the sensor responds very well to the materials having low to medium dielectric loss due to which accurate characterization of such liquids is possible.

1.1 Geometry of Sensing Element

CDR is the basic sensing structure which is made from Alumina (99.6%). The relative dielectric constant of the CDR is 9.8 and it has a dielectric loss tangent of 0.0002. The resonant frequency f_r and dimensions of CDR for $TM_{01\delta}$ resonant mode are calculated as given in (1) [12].

$$f_{r(TM_{01\delta})} = \frac{c\sqrt{3.83^2 + \left(\frac{\pi r}{2h}\right)^2}}{2\pi r\sqrt{\varepsilon_r + 2}}. \quad (1)$$

In order to get the resonance at 10.5 GHz for $TM_{01\delta}$ mode, CDR of radius r 6.35 mm and height h of 9.0 mm is used as shown in Fig. 1. CDR is placed on FR-4 substrate disc having thickness of 1.55 mm and radius of 20 mm. The CDR is fed through a probe from its bottom. The length of the probe inside CDR is optimized through simulations with the help of a commercially available finite element method based full-wave electromagnetic simulator [13]. A shallow groove is machined on the top surface of CDR so that the glass capillary containing Liquid Under Test (LUT) can be mounted on it. This arrangement was done to ensure that the capillary tube can be placed at the same location every time to ensure repeatability and validity of the results. A micro capillary made up of borosilicate glass with radius of 0.55 mm and a length l of 75 mm is used to hold LUT. The measurement arrangement is shown in Fig. 1, such arrangement requires 75 µl of LUT to fill the capillary however the actual content that is in direct interaction of electromagnetic fields is 10 µl only. In CDR different modes can be generated through different feeding techniques. In cavity perturbation theory the electromagnetic fields of the cavity are perturbed due to their interaction with the LUT therefore maximum field interaction is necessary with LUT to enforce maximum perturbation and higher sensitivity.

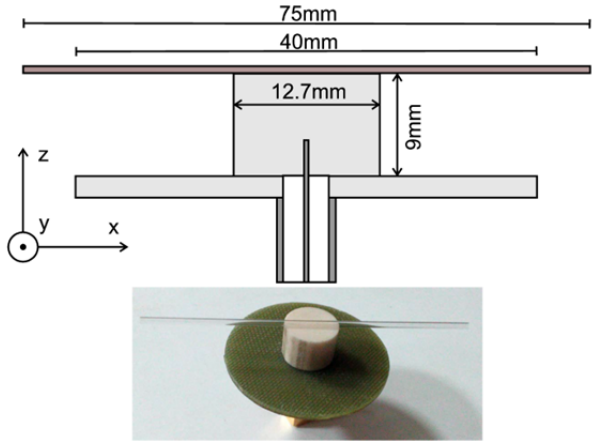

Fig. 1. Geometry of the sensor and measurement arrangement for LUT.

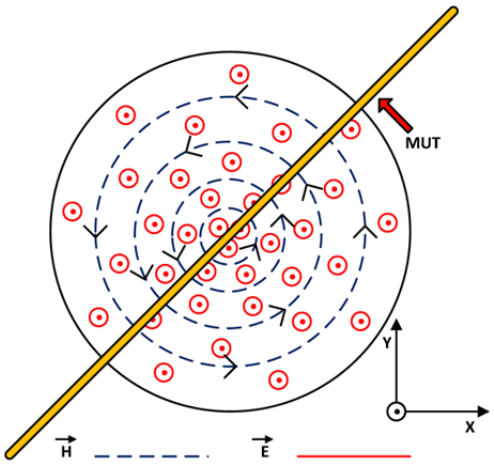

Fig. 2. Electromagnetic field distribution of $TM_{01\delta}$ DR mode.

In order to achieve large perturbation, maximum E-field is required along z axis in the center of CDR. The chosen $TM_{01\delta}$ mode offers maximum E-field in the center therefore it is launched in CDR with the help of a center fed probe. The plots of E and H-fields for $TM_{01\delta}$ are shown in Fig. 2.

1.2 Sensitivity Analysis of the Sensor

The basic objective of the proposed design is to devise a highly sensitive measurement setup, therefore to analyze the sensitivity performance a detailed analysis has been carried out. Since the perturbation in the CDR is effected by the quantity and relative permittivity of LUT therefore several simulations were carried out to analyze the variation in capillary size that is holding LUT and the relative permittivity of LUT. The resonant frequencies obtained in simulations are used to calculate fractional change F and sensitivity as given in (2), (3).

$$F = \frac{f_r - f_s}{f_s}, \quad (2)$$

$$Sensitivity = \frac{dF}{d\varepsilon'} \quad (3)$$

where f_r and f_s are the resonant frequencies with and without LUT respectively. The results obtained are presented in Figs. 3 and 4.

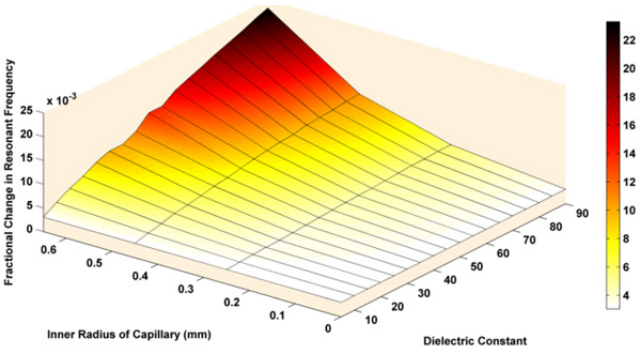

Fig. 3. Fractional change as a function of dielectric constant and capillary size.

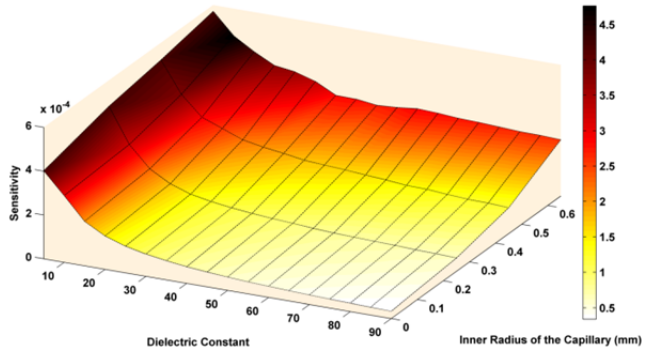

Fig. 4. Sensitivity analysis with the change in LUT quantity and dielectric constant.

It is observed in Fig. 3 that for capillary sizes of diameter 0.4 mm to 0.9 mm fractional change is linearly increasing with the increase in relative dielectric constant but for bigger diameters this increase is much larger due to higher perturbation. In the sensitivity plot shown in Fig. 4 it can be observed that the CDR is very sensitive to materials with relative dielectric constant values ranging from 1 to 30. It is also concluded that for liquids with dielectric constant above 30 the CDR is less sensitive for smaller LUT quantities. This behavior of the resonator is expected as the sensitivity is differential of fractional change with respect to dielectric constant (3) therefore for high dielectric constant materials it becomes less sensitive [4].

2. Methodology

It is evident from the sensitivity analysis that even for smaller changes in the dielectric properties of LUT there is a significant shift in the resonant frequency. This shift in resonant frequency provides information about dielectric constant ε' and loss tangent = $\varepsilon''/\varepsilon'$ of the LUT. To verify this concept with the proposed measurement setup a number of glass capillaries filled with different samples are loaded on CDR sensor to measure its behavior using a Vector Network Analyzer (VNA).

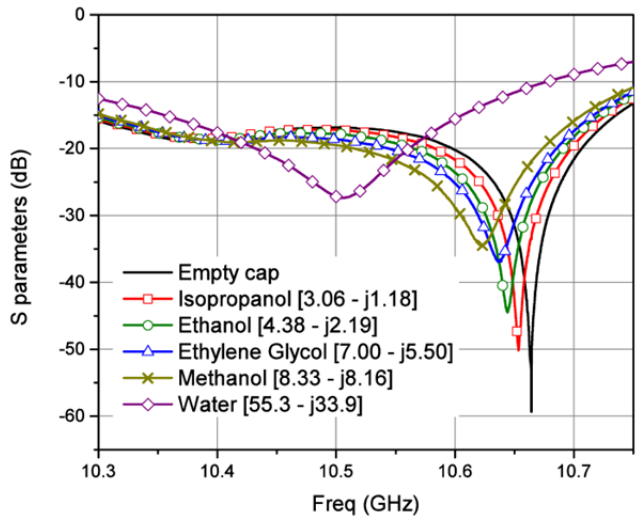

Fig. 5. Return loss vs. frequency plots for different materials.

It can be observed from the measured results shown in Fig. 5 that the CDR offers a distinguishable response for different materials. It is evident that the resonant frequency undergoes larger shifts for high permittivity liquids alongside the loss factor of these liquids affects the 3 dB bandwidth of the response. The data is measured with the average of 200 measurements in VNA to avoid any possible drift. Also the measurement campaign is run multiple times to ensure the repeatability of measurement. Since the unloaded quality factor of the structure is around 5000 which makes this sensor highly sensitive to small changes in the LUT.

The fundamental source for uncertainty in this technique is the variation among the radius of sample holding glass capillaries. Since it is established from Fig. 3 and 4 that the shift in resonant frequency increases as the radius of capillary tube increases, this count towards larger error if the radii of capillary tubes are inconsistent. The only way this error can be removed is by using high quality capillary tubes with little tolerance or making capillary part of this setup and reusing it for various samples. Another factor that produces uncertainty and error is the preparation process of reference samples. Since the quantities of mixtures are made is volume ratio there is a possibility of having variance of few micro liters this will result in error factor. However it is worth noting here that the behavior of CDR in this setup is linear therefore even if few reference samples are imperfect the linear fit relation will reduce the error. More over the volume fraction error can be reduced further by making mixtures in larger quantities.

Other factors like capillary position or orientation will not add any error factor since the $TM_{01\delta}$ mode is symmetric and it is indifferent to the orientation of the capillary tube. The position of capillary tube in the center is ensured by etching a groove on the surface so there is little change of having position offset among different sample readings.

2.1 Sample Preparation and Sensor Calibration

The initial step is to prepare known reference samples, for the proof of this investigation two types of solutions were prepared with variable concentrations. One of the solutions is Isopropanol – distilled water and the other is Ethanol – distilled water. High purity lab grade chemicals have been used for the solution preparation. The dielectric properties of the prepared solution in different concentrations are theoretically calculated by using the dielectric mixture relation as given in (4) [14].

$$\varepsilon_r(f) = \varepsilon_1(f)\left[\frac{\{2\varepsilon_1(f)+\varepsilon_2(f)\}+2q\{\varepsilon_2(f)-\varepsilon_1(f)\}}{\{2\varepsilon_1(f)+\varepsilon_2(f)\}-q\{\varepsilon_2(f)-\varepsilon_1(f)\}}\right] \quad (4)$$

where $\varepsilon_r(f)$ is the complex permittivity of solution at a specific frequency, $\varepsilon_1(f)$ is a complex permittivity of entire matrix which in this case is isopropanol or ethanol, $\varepsilon_2(f)$ is the complex permittivity of the inclusion which in this case is distilled water and q is the volume fraction of the solu-

tion. Values of q ranges from 0 to 1, for pure isopropanol or ethanol (entire matrix) with no water (inclusion) included the value of q is 0 and it changes with the varying amount of water being added in the matrix to the maximum value of 1, which means only inclusion. The values of complex permittivity of pure liquids at the frequency of operation are taken from the tables of dielectric dispersion [15]. At 10.5 GHz complex permittivity of isopropanol is $3.06 - j1.18$, for ethanol it is $4.38 - j2.19$ and for water it is $55.3 - j33.9$. For both the solutions the calculated values from the mixer theory for permittivity and loss tangent are plotted in Fig. 6 for different values of q.

The graphs given in Fig. 6 provide the relations of permittivity and loss tangent as a function of volume fraction q through interpolation of data. The computed relations from Fig. 6 for dielectric constant of isopropanol – water solution, loss tangent of isopropanol – water solution, dielectric constant of ethanol – water solution and loss tangent of ethanol – water solution are given in (5), (6), (7), and (8) respectively.

$$\varepsilon_r = 18q^4 - 1.53q^3 + 8.14q^2 + 7.78q + 3.06, \quad (5)$$

$$\tan\delta = 0.013q^4 + 0.089q^3 - 0.053q^2 + 0.077q + 0.386, \quad (6)$$

$$\varepsilon_r = 17.44q^4 - 0.003241q^3 + 9.632q^2 + 10.38q + 4.38, \quad (7)$$

$$\tan\delta = -0.0046q^4 + 0.066q^3 - 0.0385q^2 + 0.055q + 0.5. \quad (8)$$

Once the calibration samples are prepared they are mounted on the sensor and measured through VNA. It is observed that each sample shifts the resonant frequency of CDR to a certain value. This shift in the resonant frequency Δf is linearly related to the change in dielectric constant and 3-dB bandwidth BW is linearly related to the loss tangent of the LUT. Therefore, the shift in resonant frequency and 3 dB BW are calibrated against known samples of the solution. The calibration graphs for isopropanol – water solutions and ethanol – water solutions are shown in Figs. 7 and 8, respectively. The linear behavior observed from Figs. 7b, 7c and Figs. 8b, 8c are in accordance with the resonant perturbation theory. In isopropanol – water solution the maximum error of 3.2% and 2.4% has been occurred in volume fraction from resonant shift and 3 dB bandwidth respectively. Likewise in ethanol – water solution the maximum error of 2.9% and 1.5% has been noted from resonant shift and 3 dB bandwidth respectively. The relations given in (9) and (10) are empirically calculated from these graphs for isopropanol – water and ethanol – water solution respectively.

$$\left.\begin{aligned} q &= 0.011\Delta f - 0.088 \\ q &= 0.0119BW - 0.0142 \end{aligned}\right] \quad (9)$$

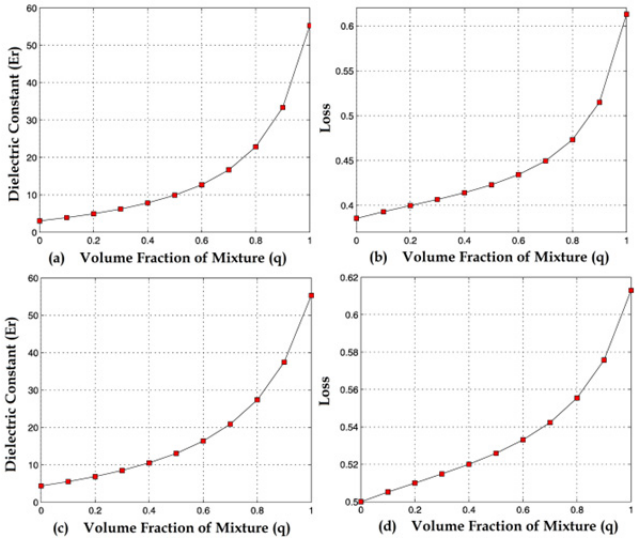

Fig. 6. Theoretically calculated values for different volume fraction (a) Dielectric constant of isopropanol water solution. (b) Loss tangent of isopropanol water solution. (c) Dielectric constant of ethanol water solution. (d) Loss tangent of ethanol water solution.

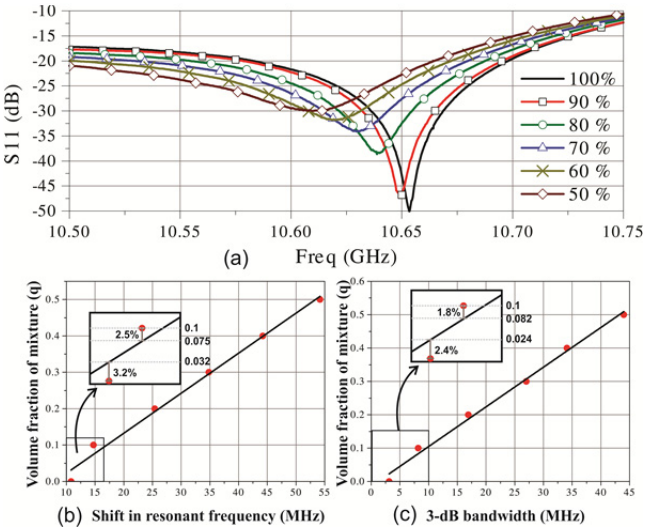

Fig. 7. Measured response for the solution of isopropanol and distilled water: (a) Return loss vs. frequency for different concentration of solutions. (b) Change in resonant frequency Δfvs. volume fraction of solution. (c) 3-dB bandwidth BW vs. volume fraction of solution.

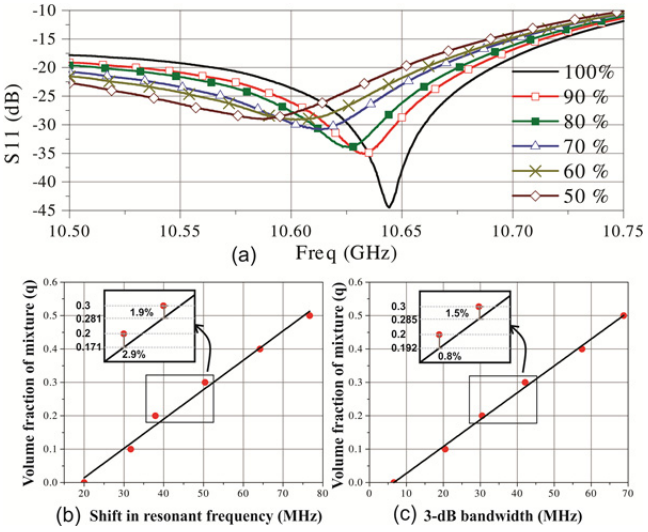

Fig. 8. Measured response for the solution of ethanol and distilled water: (a) Return loss vs. frequency for different concentration of solutions. (b) Change in resonant frequency Δf vs. volume fraction of solution. (c) 3-dB bandwidth BW vs. volume fraction of solution.

$$\left.\begin{aligned} q &= 0.088\Delta f - 0.16 \\ q &= 0.00805BW - 0.0532 \end{aligned}\right]. \quad (10)$$

It is also evident from Figs. 7 and 8 that resonant frequency decreases with increased volume of water mixed in isopropanol and ethanol. This behavior is due to the fact that the permittivity of water is much higher than that of isopropanol and ethanol therefore with the increased volume of water overall permittivity of the solution increases which results in the decrease of resonant frequency. It is observed that for the case of isopropanol – water solution the change of 0.5 in dielectric constant gives a shift of 3.3 MHz in the resonant frequency and a change of 0.5 in loss tangent gives a change of 129 MHz in 3-dB bandwidth. For the case of ethanol and water solution it is observed that the change of 0.5 in dielectric constant offers resonant frequency shift of 3.3 MHz and a change of 0.1 in loss tangent gives a change of 245 MHz in the 3-dB bandwidth. It shows very high sensitivity of the sensor with which very small changes in the mixture concentrations can be measured.

For measurement of unknown samples of isopropanol-water solution or ethanol – water solution the LUT is mounted on the sensor and the resonant shift or 3-dB bandwidth is measured from VNA. Both of these values correspond to the volume fraction of the unknown LUT through relations (9) or (10). These volume fractions are used in mathematical relations (5)–(8) to calculate the dielectric constant and loss tangent of the unknown samples. The whole process of calibration of a new solution and measuring an unknown sample is shown in the flow chart given in Fig. 9.

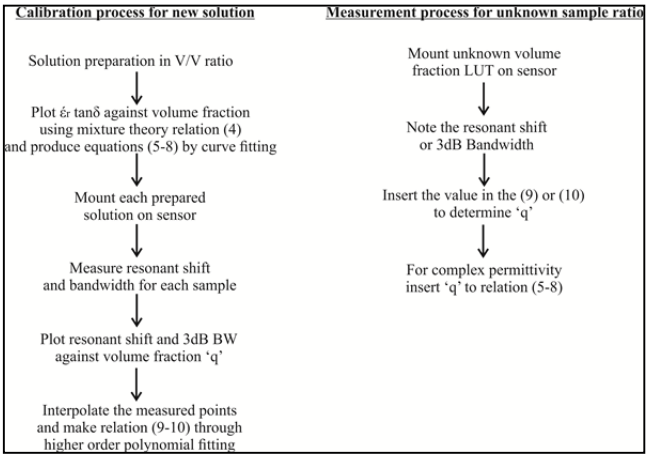

Fig. 9. Flow chart for calibration process and measurement process of unknown LUT.

2.2 Measured Results and Comparison

A number of samples from isopropanol – water solution and ethanol – water solution with random volume fractions have been measured using the proposed sensor. The measured values are used for estimating volume fraction and ultimately the dielectric constant and loss tangent of the LUT. In Tabs. 1 and 2, measured results obtained through CDR are compared with the theoretical results computed with mixture theory.

Volume fraction q	Relative dielectric constant ε_r'			Loss tangent		
	Mixture theory [14]	$TM_{01\delta}$ mode CDR	Error (%)	Mixture theory [14]	$TM_{01\delta}$ mode CDR	Error (%)
0	3.06	3.30	7.84	0.3901	0.3877	0.61
0.1	3.92	3.79	3.32	0.3929	0.3922	0.18
0.2	4.95	4.86	1.82	0.3997	0.3992	0.12
0.3	6.23	6.16	1.12	0.4063	0.4075	0.29
0.4	7.83	7.81	0.25	0.4142	0.4139	0.07
0.5	9.91	10.12	2.12	0.4230	0.4243	0.30

Tab. 1. Comparison of measured results with theoretical data for isopropanol – water solution.

Volume fraction q	Relative dielectric constant ε_r'			Loss tangent		
	Mixture theory [14]	$TM_{01\delta}$ mode CDR	Error (%)	Mixture theory [14]	$TM_{01\delta}$ mode CDR	Error (%)
0	4.38	4.53	3.42	0.5000	0.5001	0.02
0.1	5.52	5.73	4.79	0.5050	0.5058	0.16
0.2	6.87	6.46	9.36	0.5099	0.5097	0.04
0.3	8.50	8.18	7.3	0.5148	0.5141	0.13
0.4	10.52	10.58	1.36	0.5199	0.5205	0.11
0.5	13.07	13.45	8.67	0.5259	0.5260	0.02

Tab. 2. Comparison of measured results with theoretical data for ethanol – water solution add error %.

It is observed that the measured results are in good agreement with the theoretical values of dielectric constant and loss tangent. The maximum measured dielectric constant errors of 10.1% and 13.4% have been observed for isopropanol–water and ethanol–water solutions respectively. The error percentage increase with the value of dielectric constant which is predicted by Figs. 3 and 4, that shows the higher sensitivities for high dielectric constant values. In case of loss tangent the errors are considerably smaller and the maximum errors of 0.61% and 0.16% have been measured for isopropanol–water and ethanol–water solution respectively. The error came up during measurement reported in Tab. 1 and 2 are cumulative errors from curve fitting in (5)–(10), this will also involve uncertainty in the mixture theory curves. These results clearly show that the CDR sensor is highly sensitive to small changes in the solution concentrations.

3. Conclusion

A very high Q top loaded $TM_{01\delta}$ mode cylindrical dielectric resonator is proposed in this work which is highly sensitive and compact. Owing to top loading option of LUT it is much easier to handle as compared to closed cavity models or waveguide characterization techniques. It is shown that the quantities of liquid as small as 10 µl are sufficient to characterize their complex permittivity. The

characterization of isopropanol–water solutions and ethanol–water solutions have been carried out and compared with the results obtained from the mixture theory. An excellent agreement has been observed with the theoretical results. The process can be extended to all low to medium loss liquid solutions. The sensitivity and robustness of the technique make it a decent choice to be used for liquid characterization in pharmaceutical industry.

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